MASTER (MA)

Films, Foams & Monoliths: Novel Forming Methods for Hydrogen Adsorbent Composites

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Award date:
2019

Awarding institution:
University of Bath

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Novel Forming Methods for Hydrogen Adsorbent Composites

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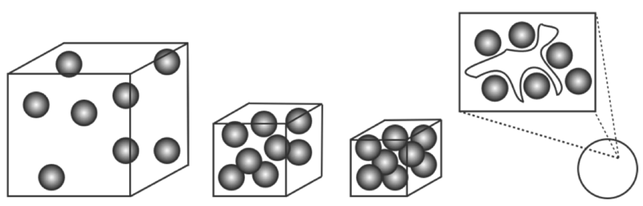
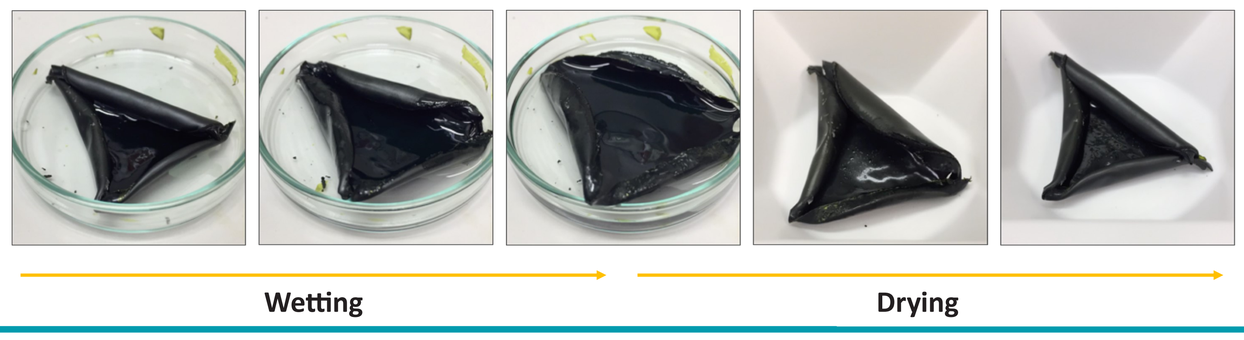
Introduction & Objectives

Several **high surface area materials** have been developed in respect to **hydrogen storage**. However, efforts have thus far **focused on hydrogen uptake alone**. Without due attention to the **relevant mechanical properties** required for realistic application in hydrogen compression vessels, it is unlikely that the U.S. Department of Energy **target of 5.5% wt. storage by 2050** will be attained.



Atmospheric	Gen 1 Comp.	Gen 2 Comp.	Porous	Liquid	Water
1 Bar	300 Bar	700 Bar	≤	20 K	Ambient
0.04 MJ/L	3.98 MJ/L	5.68 MJ/L	9.94 MJ/L	10.08 MJ/L	15.76 MJ/L

Fig 1. *Hydrogen storage methods and corresponding conditions and volumetric energy densities. Values from [1].*



Powders & Films

The polymer of intrinsic microporosity, **PIM-1**, has a **permanent porosity ideal for hydrogen storage**, and is solution processable in chloroform to form thin films. However, it has a **relatively low surface area (~ 800 m^2 g^{-1})**, limiting the number of adsorption sites. This can be improved by the **incorporation of the particulate filler, AX21** (~3000 m^2 g^{-1}), during film casting.

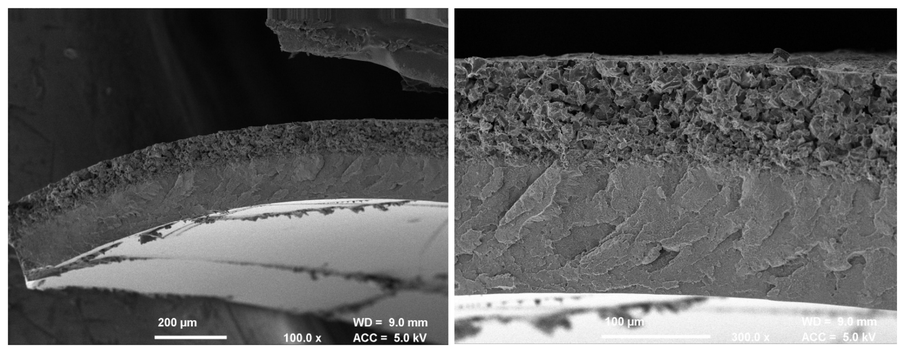


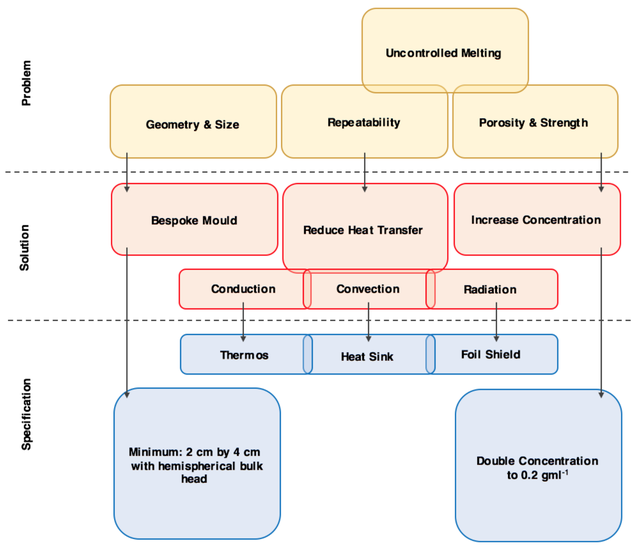
Fig 2. *(Right above) SEM micrographs of composite film displaying bilayer effect.*

(Right below) Flexibility of PIM-1 films.

(Left) Reversible actuation of composite films in smart response to organic solvents.

Foams & Method Development

Freeze casting is a forming method suited to materials incompatible with standard plastic thermoforming techniques such as injection moulding or crosslinking. By **directionally freezing** solutions (0.1 g ml^{-1}), PIM-1 **foams containing solvent template microstructures** have been achieved. However, these foams were **too brittle to handle**, and thus did not meet the project goals. The **method was developed** following the matrix below:



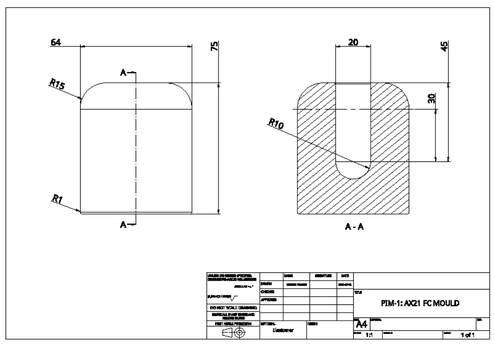
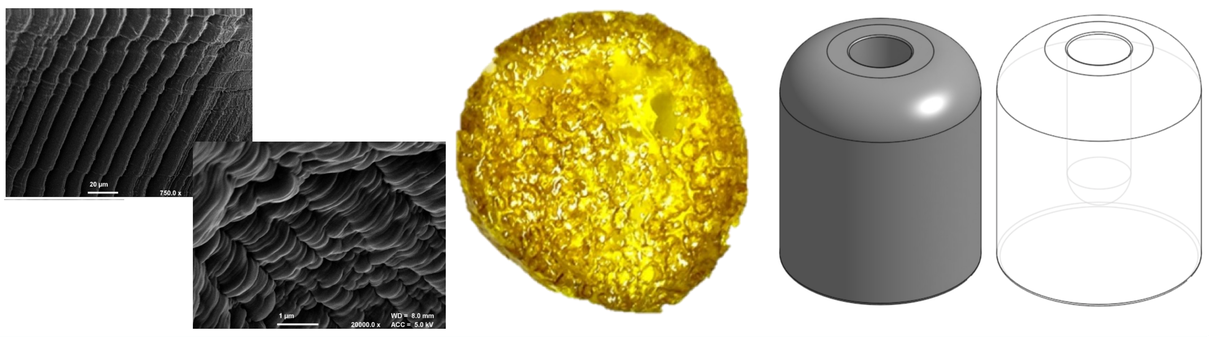


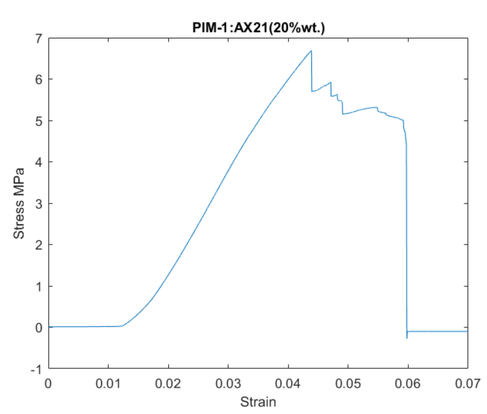
Fig 3. *(Left) Problem matrix for method and solutions.*
(Right) SEM micrographs evidence solvent templating effect at multiple scales in...
(Adjacent) ...PIM-1 foams.
(Above) Elastic mould design .



Monoliths

The **novel methodology was successful** at creating monoliths of a scaled-up size and predefined geometry, resulted in mechanical characteristics improved beyond that possible with traditional freeze casting techniques. As the **strain to failure was > 0.019**, these monoliths could be feasibly **employed in *Type IV* (70 MPa) hydrogen compression tanks**, without detriment to safety. [2]

The inclusion of **20% wt. AX21 enhanced the BET surface area** of the monolith above that of pristine PIM, from **762—849 m^2 g^{-1}**, with scope to increase this further.



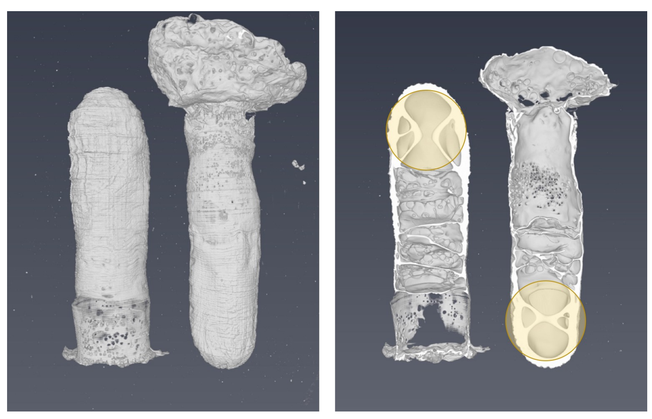


Fig 4. *(Right) CT rendering of monolith cross section show anisotropic void fraction.*
(Above) Compression testing results for composite monolith demonstrating 'graceful' failure.
(Left) Compression test samples and demonstration of the ease by which they can be handled.

Conclusions & Future Work

- A **novel freeze casting method** has been developed to produce **hydrogen adsorbent monoliths of predictable size and geometry**. The method could foreseeably be **adopted by industry** simply by adapting existing freeze casting procedures. By **introducing ease of handling, as well as heat and mass transfer characteristics**, the applications for PIM-1 have been broadened beyond that of powders and films.
- Composite monoliths had **enhanced BET surface areas**, with scope to increase this further. Monoliths also had **sufficient strength for safe application in compression tanks**.
- It is postulated that **several composite monolith variants** may be possible, where high surface area fillers such as **MOFs, COFS, PAFs and other carbons could be explored.**

[1] A. M. Abdalla, S. Hossain, O. B. Nisfindy, A. T. Azad, M. Dawood and A. K. Azad, *Energy Convers. Manage.*, 2018, **165**, 602-627.

[2] K. Polak-Krasna, R. Dawson, L. T. Holyfield, C. R. Bowen, A. D. Burrows and T. J. Mays, *J. Mater. Sci.*, 2017, **52**, 3862-3875.

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